DZS 1236: 2022
ICS 75.160
FIRST EDITION

Draft Zambian Standard

Denatured Hydrous Ethanol for Use as Cooking and Appliance Fuel – Specification

ZAMBIA BUREAU OF STANDARDS

Amendments issued since publication

Amdt No.	Date	Text affected

DATE OF PUBLICATION

This Zambian Standard has been prepared and published under the authority of the Zambia Bureau of Standards on ……..

ZAMBIA BUREAU OF STANDARDS

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A Zambian standard does not purport to include all the necessary provisions of a contract. Users of Zambian standards are responsible for their correct application.

TECHNICAL COMMITTEE RESPONSIBLE

This Draft Zambian Standard was prepared by the Renewable Energy Technical Committee, (TC 4/20), upon which the following organizations were represented:

Biofuels Association of Zambia
Energy Regulation Board
Ministry of Energy
Sunbird bioenergy Zambia Limited
Tapera Industries Limited
Thomro Investments Limited
University of Zambia – School of Engineering
United States Agency for International Development – Alternatives to Charcoal Project
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FOREWORD

The Zambia Bureau of Standards (ZABS) is the Statutory Organization established by an Act of Parliament. ZABS is responsible for the preparation of national standards through its various Technical committees composed of representation from government departments, the industry, academia, regulators, consumer associations and non-governmental organizations.

This Draft Zambian Standard has been prepared with assistance drawn from: ASTM E3050 – 16 "*Standard Specification for Denatured Ethanol for Use as Cooking and Appliance Fuel Standards*", published by ASTM International and in accordance with the procedures of ZABS. All users should ensure that they have the latest edition of this publication as standards are revised from time to time.

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The preparation of this Draft Standard has been undertaken by the Renewable Energy Technical Committee, (TC 4/20).

ACKNOWLEDGEMENT

The Zambia Bureau of Standards would like to acknowledge the invaluable support of the United States Agency for International Development (USAID), Energy Regulation Board (ERB) and all the institutions and stakeholders that contributed in the promulgation of this standard.

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ZAMBIA BUREAU OF STANDARDS

Draft Zambian Standard

Denatured Hydrous Ethanol for Use as Cooking and Appliance Fuel – Specification

1.0 SCOPE

This Draft Zambian Standard specifies requirements, sampling and test methods for denatured hydrous ethanol for use as cooking, heating and any other burning appliances.

2.0 NORMATIVE REFERENCES

The under listed documents contain provisions which, through reference in this text, constitute provisions of this standard. All documents are subject to revision and, since any reference to a document is deemed to be the latest reference to the latest edition of that document, parties to agreements based on this standard are encouraged to take steps to ensure the use of the most recent editions of the documents indicated below. Information on currently valid national and international standards may be obtained from the Zambia Bureau of Standards:

ASTM D203	Test Method for Water Using Volumetric Karl Fischer Titration
ASTM E300	Standard Practice For Sampling Industrial Chemicals
ASTM E1064	Test Method for Water in Organic Liquids by Coulometric Karl Fischer Titration
ASTM D1353	Standard Test Method for Nonvolatile Matter in Volatile Solvents for Use in Paint, Varnish, Lacquer, and Related Products
ASTM D4057	Standard Practice for Manual Sampling of Petroleum and Petroleum Products
ASTM D4177	Standard Practice for Automatic Sampling of Petroleum and Petroleum Products
ASTM D4306	Standard Practice for Aviation Fuel Sample Containers for Tests Affected by Trace Contamination
ASTM D4815	Test Method for Determination of MTBE, ETBE, TAME, DIPE, tertiary-Amyl Alcohol and C1 to C4 Alcohols in Gasoline by Gas Chromatography
ASTM D5501	Test Method for Determination of Ethanol and Methanol Content in Fuels Containing Greater than 20% Ethanol by Gas Chromatography
ASTM D5854	Standard Practice for Mixing and Handling of Liquid Samples of Petroleum and Petroleum Products
ASTM D6045	Standard Test Method for Colour of Petroleum Products by the Automatic Tristimulus Method, published by ASTM International.
ASTM D6839	Standard Test Method for Hydrocarbon Types, Oxygenated Compounds, Benzene, and Toluene in Spark Ignition Engine Fuels by Mulitidimensional Gas Chromatography
ASTM D7795	Standard Test Method for Acidity in Ethanol and Ethanol Blends by Titration
ISO 10101-2	Natural gas - Determination of water by the Karl Fischer method - Part 2: Titration procedure.
ISO 10101-3:	Natural gas - Determination of water by the Karl Fischer method - Part 3: colometric procedure.
ISO 1388-2	Ethanol for industrial use – Methods of test – Part 2: Detection of alkalinity or acidity to phenolphthalein.
ISO 22854	Liquid petroleum products - Determination of hydrocarbon types and oxygenates in automotive-motor gasoline and in ethanol (E85) automotive fuel - Multidimensional gas chromatography method

3.0 TERMINOLOGY

For the purpose of this Standard, the following terms and definitions shall apply.

3.1. **Denatured ethanol:** ethanol made unfit for beverage use by the addition of toxic or noxious materials.

3.2. **Hydrous ethanol:** n-ethyl alcohol, the chemical compound C_2H_5OH.

3.3. **Higher alcohols:** aliphatic alcohols of general formula $C_nH_{2n}+1OH$ with n being 3 to 8.

3.4. **Hydrocarbon:** components in an ethanol-hydrocarbon blend containing only hydrogen and carbon.

4.0 REQUIREMENTS

4.1. General requirements

4.1.1. The product shall be visually free of sediment and suspended matter.

4.1.2. The product shall be free of any adulterant or contaminant that can render the material unacceptable for its commonly used applications.

4.1.3. The product shall be colored to visually indicate that it is not potable.

4.2. Specific requirements

The product when tested according to the methods prescribed, shall comply with the specific requirements given in Table 1.

Table 1: Specific requirements for Denatured Hydrous Ethanol.

S/No.	Characteristic	Requirement	Test method
i.	Ethanol content, % volume, Min	90	ASTM D5501
ii.	Water content, % by volume , Max	10	ASTM E203, ASTM E1064, ISO 10101-2 or ISO 10101-3:
iii.	Higher Alcohols (C_3–C_8), volume %, Max	2	ASTM D4815 or ISO 1388-2
iv.	Hydrocarbon[1] , % volume, Max	1	ASTM D6839 or ISO 22854
v.	Acidity (as acetic acid), mg/kg, Max	40	ASTM D7795 or ISO 1388-2
vi.	Denatonium Benzoate, mg/Kg, (Min–Max)	10 - 20	Annex A
vii.	Colored Dye, mg/Kg, Max	10	ASTM D6045
viii.	Residue on evaporation, %, m/v, max.	0.01	ASTM D1353
Note 1: The hydrocarbons presence under this specification are as follows: gasoline, unleaded gasoline, natural gasoline, heptane, or rubber hydrocarbon solvent. Note 2: For examples of recommended denaturants, refer to Annex B			

5.0 PACKAGING AND LABELLING

5.1. Packaging.

5.1.1 The product shall be supplied and stored in clean, dry and tight containers, without faults, made of material which does not react with alcohol. The method of closing the containers shall prevent the contents from contamination and evaporation.

5.1.2 All storage containers should be evaluated for durability, compatibility, and contamination of cooking and appliance fuel prior to use. If metal containers are used, do not use soldered metal containers. Soldering flux in the containers and the lead in the solder can contaminate the product.

5.2. Labelling

Each container shall bear the following information given in prominent, legible and indelible mark:

a) Name of the product as "Denatured Hydrous Ethanol"
b) Manufacturer's name and address.
c) Recognized trademark, if any.
d) Intended use.
e) Name(s) of denaturant
f) Nominal content
g) Nominal strength.
h) Batch/lot number.
i) Manufacturing date
j) Pictorial symbols to show flammability and toxicity nature.

Figure 1: Flammability

Figure 2: Toxicity

k) Instruction of use
l) Storage instructions

6.0 SAMPLING AND TESTING

6.1. Sampling

The user is strongly advised to review all intended test methods prior to sampling to understand the importance and effects of sampling technique, proper containers, and special handling required for each test method.

6.1.1 Correct sampling procedures are critical to obtain a sample representative of the batch intended to be tested. Care shall be taken to use appropriate procedures for manual method sampling and automatic method sampling, as applicable. (Refer to ASTM D4057 or ASTM E300 and ASTM D4177)

6.1.2 The correct sample volume and appropriate container selection are important decisions that can impact test results. (Refer to ASTM D4306 and ASTM D5854 for procedures on container selection and sample mixing and handling)

6.1.3. All sampling and storage containers should be evaluated for durability, compatibility, and contamination of cooking and appliance fuel prior to use.

6.1.4 If samples must be collected in metal containers, do not use soldered metal containers. Soldering flux in the containers and the lead in the solder can contaminate the sample.

6.1.5 Sample *Size*—A minimum of about 1 L is recommended.

6.1.6 Batch Size— A *batch* shall normally consist of the amount contained in a storage tank or other bulk container. If this definition does not apply, the definition of a batch must be agreed upon between the supplier and purchaser.

6.2. Testing

6.2.1. Tests shall be carried out according to the methods prescribed in Table 1 of this standard.

7.0 CERTIFICATION

Certification shall be conducted by Zambia Bureau of Standards or any competent body, public or private and the ZABS mark of quality shall be awarded as proof of certification.

Figure 3: ZABS Certification mark

ANNEX A
(Normative)
Determination of Denatonium Benzoate in Alcohol Products by HPLC-UV

A.1. Principles

This document describes a standard method for the determination of denatonium benzoate (DB) in Completely Denatured Alcohol (CDA) formulations using HPLC with UV detection at 210 nm. The samples are directly injected into the HPLC system after membrane filtration. The working range for quantitative determination of DB is 0.5 to 20.0 mg / L.

A.2. Instrumentation and materials

A.2.1. HPLC system equipped with.

- Pumping system suitable for isocratic elution.
- Solvent degassing system (on-line/off-line).
- Injection system with 20 µl loop.
- Analytical column, for example: LiChrospher 100 CN (5 µm) in LiChroCART 250-4 guard column
- Thermostated column compartment (oven).
- Diode array detector (DAD) or UV detector.
- Integrator or computer with data acquisition software and printer.

A.2.2. Analytical balance with a precision of 0.1 mg.

A.2.3. 0.5, 2, 5, 10 and 20 ml pipettes.

A.2.4. 100 and 1000 ml volumetric flasks.

A.2.5. Weighing bottle.

A.2.6. Syringes.

A.2.7. 0.45 µm cellulose membrane filters.

A.2.8. 2.8 250 ml beaker.

A.2.9. 1000 ml graduated cylinder.

With regard to the HPLC column, the recommended analytical column in this method is LiChrospher 100 CN (5 µm) as described above. However, alternative HPLC columns (C_{18} / C_8), buffers and chromatographic parameters may be used provided that good peak shape is obtained for denatonium benzoate and good separation of denatonium benzoate from potential interferences can be achieved.

A.3. Reagents and solutions.

A.3.1. Denatonium benzoate, purity ≥ 99 %. Handle it with gloves.

A.3.2. Ethanol 96 % vol.

A.3.3. Sodium chloride, extrapure.

A.3.4. Acetonitrile, HPLC grade.

A.3.5. Water, HPLC grade.

A.3.6. 0.2 % sodium chloride solution.
Weigh 0.4 g of sodium chloride in a weighing bottle and dissolve it in a beaker with 200 ml of water HPLC grade.

A.3.7. Mobile phase
Add in a 1000 ml volumetric flask, 200 ml of 0.2 % sodium chloride solution and 800 ml of acetonitrile HPLC grade.

A.4. Standard solutions

A.4.1. Preparation of the stock solution (100 mg DB / L).

Weigh, recording the exact weight, 0.1 g of denatonium benzoate in a weighing bottle and dissolve it in a 1000 ml volumetric flask with ethanol 96 % vol. Mix gently.

Measure the mass of this solution with a top loading balance and the density at 20ºC with an electronic densimeter.

A.4.2. Preparation of the working calibration solutions.

Add, in 100 ml volumetric flasks, 20 ml of ethanol 96 % vol. (to minimize weighing errors), then 0.5, 2, 5, 10 or 20 ml (weighing) of the stock solution and top up to the filling mark with ethanol 96 % vol. Mix gently.

A.5. Chromatographic and calibration parameters.

When using LiChrospher 100 CN (5 µm) column, chromatographic and calibration parameters recommended are:

- Column flow: 1.2 ml / min.
- Stoptime: 14 min.
- Detector: signal 210 nm (bandwidth 8 nm), reference 360 nm (bandwidth 100 nm).
- Mobile phase: acetonitrile 80:20 0.2 % sodium chloride solution.
- Injection volume: 20 µl.
- Column oven temperature: 27ºC.
- Calibration: external standard.
- Signal: peak area.
- Curve type: linear.
- Origin: included.
- Weight: equal.

A.6. Calibration.

Working solutions containing the following concentrations of denatonium benzoate 0.5, 2, 5, 10 and 20 mg / l are analysed by injecting one replicate of each working solution. Peak areas corresponding to denatonium benzoate are plotted according to the respective concentrations in order to obtain a linear regression line expressed by the formula $y = ax + b$. The correlation coefficient must be > 0.99. Otherwise, the system must be checked to improve the linear regression if possible, or the working solutions must be discarded and a new set of calibration solutions should be prepared.

A.7. Analysis of samples

No specific sample preparation is required. The samples are directly injected into the HPLC system after 0.45 µm cellulose membrane filtration.

A.8. Instrument Results:

The instrument denatonium benzoate results are calculated by comparing the sample denatonium peak area response to the calibration curve for the denatonium benzoate. This calibration curve is part of the instrument method.

ANNEX B
(Informative)
Examples of recommended denaturants

1. Benzol
2. Denatonium benzoate
3. Ether
4. Gasoline
5. Methanol
6. Methyl isobutyl ketone
7. Pyridine or Brucine
8. Tert-butanol